

PATENT SPECIFICATION

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(54) METHOD OF AND APPARATUS FOR ENRICHING SPECIMENS FOR TRACE ANALYSIS BY X-RAY FLUORESCENCE

(71) We, GESELLSCHAFT FÜR KER-
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 SCHIFFFAHRT MBH, a Body Corporate
 organised under the laws of the Federal
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 D-2054 Geesthacht-Tesperhude, Federal
 Republic of Germany, do hereby declare
 the invention for which we pray that a
 patent may be granted to us and the method
 by which it is to be performed, to be
 particularly described in and by the follow-
 ing Statement:—

This invention relates to a method of
 enriching specimens for subsequent trace
 analysis by X-ray fluorescence with a
 totally reflecting specimen carrier having a
 plane surface.

With the known methods of enrichment,
 that is to say increasing the concentration
 in the specimen of the trace substance under
 investigation, considerable difficulties were
 involved in reducing the detection limit
 during the trace analysis. These difficulties
 lay, above all, in the fact that special vessels
 were used, the contents of which had to be
 transferred to the specimen carrier after a
 chemical reaction had been effected. Only
 relatively large specimen amounts could be
 treated in these special vessels, and these
 amounts were often not available, nor were
 they need during subsequent analysis. Furthermore, losses of trace elements re-
 sulted from the influence of the surface of
 the reaction vessel on the specimen and con-
 tamination also occurred during the transfer
 of the enriched specimen from the reaction
 vessel to the specimen carrier.

It is an object of the present invention to
 provide an improved method of enriching
 specimens which solves the problem of
 applying the enriched specimen to the
 specimen carrier.

Accordingly, the present invention con-
 sists in a method of enriching specimens
 for subsequent trace analysis by X-ray

fluorescence with a totally reflecting speci-
 men carrier having a plane surface,
 characterised in that a specimen for en-
 riching is applied directly to the specimen
 carrier spaced from the margin thereof,
 appropriate techniques of enriching the
 specimen being then employed.

The invention is explained in detail be-
 low by way of example with reference to
 the accompanying drawing in which:—

Figure 1 shows a perspective illustration
 of a specimen carrier with material to be
 analyzed, and

Figure 2 shows a reaction chamber for
 the direct treatment of specimens on speci-
 men carriers for X-ray fluorescence analysis.

The specimen carrier of an associated
 X-ray fluorescence device (as shown for
 example in DT-OS 26 32 001) illustrated in
 Figure 1, consists of a plate 1 having a
 plane-ground surface of about 10 cm² of
 quartz or another suitable material. The
 specimen material 2, which is not yet
 enriched, is applied to the surface of the
 plate 1 so that at most an area of 1 cm²
 in the centre is covered.

An oxidation of the specimen material 2
 is carried out on this specimen carrier 1.
 The specimen on the specimen carrier is
 exposed, as required, to high or low pressure
 and temperatures, suitable electromagnetic
 fields and a suitable gaseous environment.
 The specimen on the specimen carrier may
 also be treated with small amounts of suit-
 able reagents. If the specimen carrier is
 first cooled, the amount of specimen which
 can be applied may be increased if
 necessary. The specimens on the specimen
 carriers may also be freeze-dried.

As Figure 2 shows, no difficulties are in-
 volved in treating the specimen carrier with
 material to be analyzed. The specimen
 carriers, possibly in a plurality, are deposited
 on the working plate 3 of a substantially
 gas-tight reaction chamber 4 which may be

provided with a heating or cooling connection 5, gas supply 6, gas outlet 7, vacuum connection 8 and electrodes 9 for an electrical field, for the reactions to be carried out.

The method according to the invention avoids the abovementioned disadvantages and renders it possible to increase considerably the sensitivity which can be achieved with an X-ray fluorescence analysis device. By the method according to the invention, it is possible to work with the smallest amounts of specimen of a few milligrams, which are sufficient for the said analysis devices. The very small amounts of specimen which can be used also lead to a considerable shortening of the reaction times. A further advantage is to be seen in the fact that any chemical reactions which may have taken place incompletely or any insoluble components have substantially no adverse effect on the method because the reaction products remain *in situ* in any case and do

not have to be picked up again.

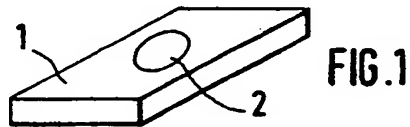
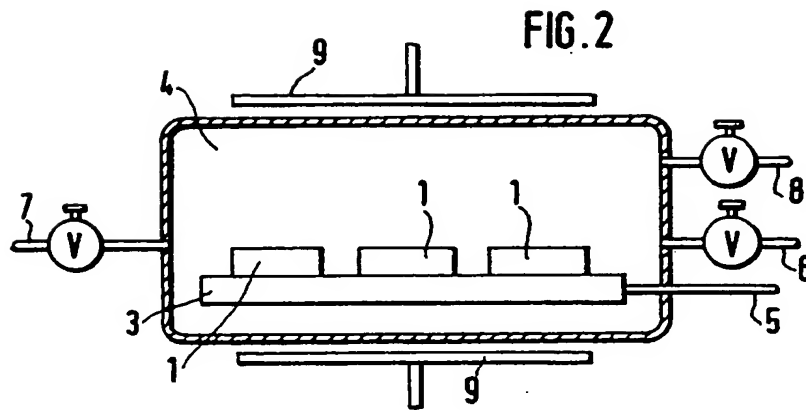
WHAT WE CLAIM IS:—

1. A method of enriching specimens for subsequent trace analysis by X-ray fluorescence with a totally reflecting specimen carrier having a plane surface, characterised in that a specimen for enriching is applied directly to the specimen carrier spaced from the margin thereof, appropriate techniques of enriching the specimen being then employed.

2. A method of enriching specimens for subsequent trace analysis by X-ray fluorescence with a totally reflecting specimen carrier having a plane surface, substantially as herein before described with reference to the accompanying drawings.

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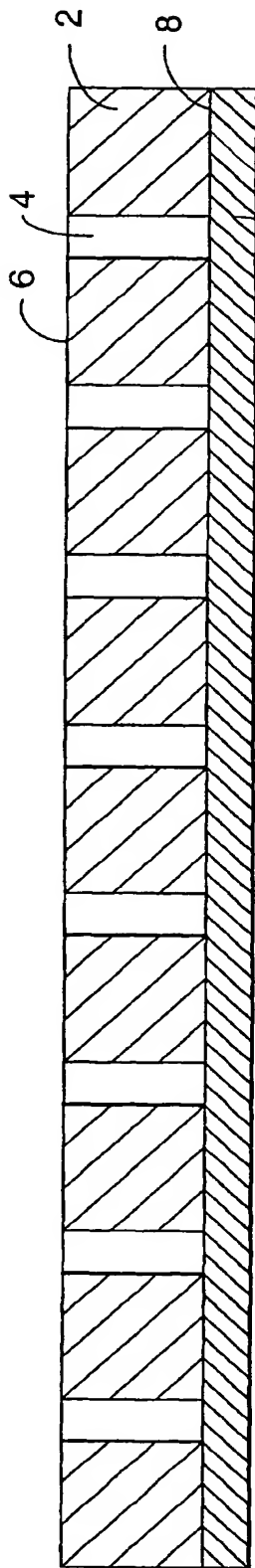


Fig. 1

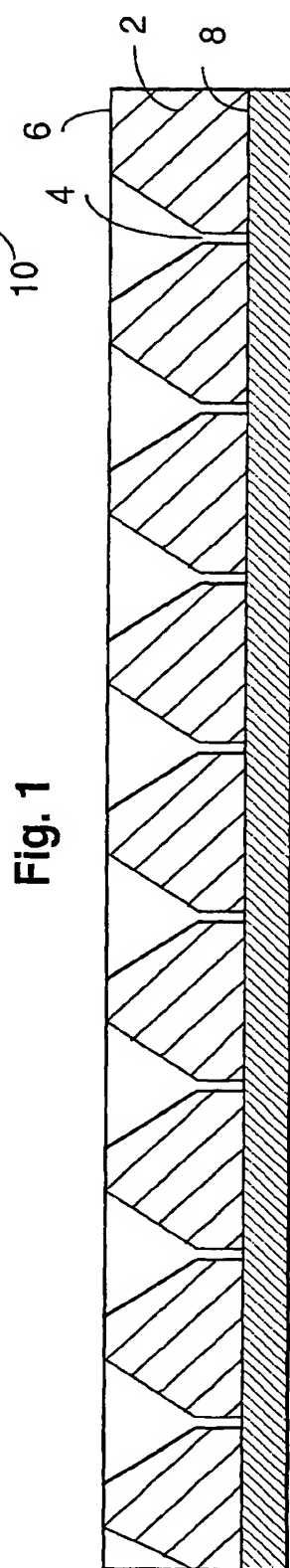


Fig. 2

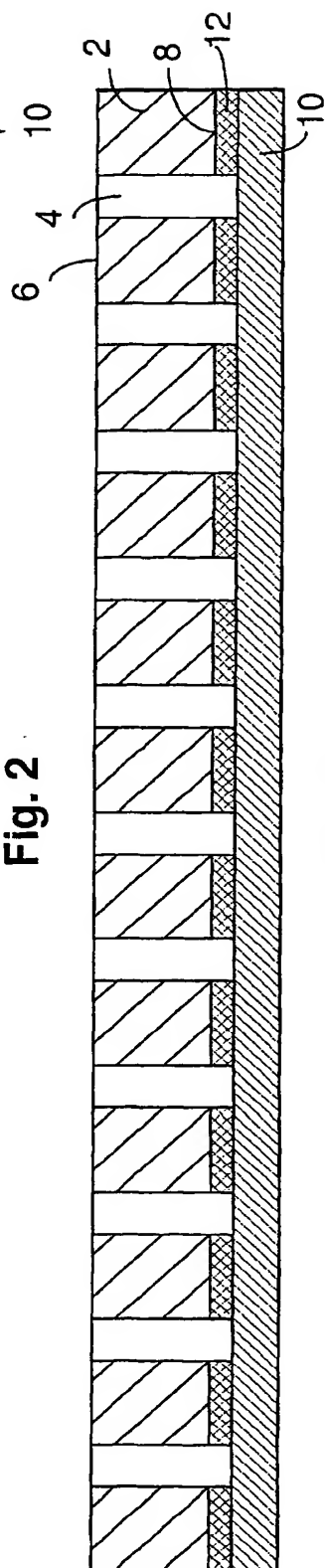


Fig. 3

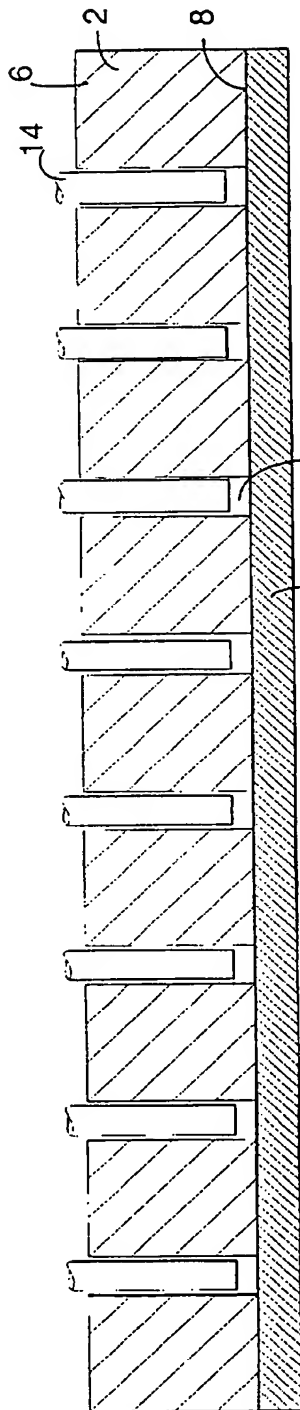


Fig. 4

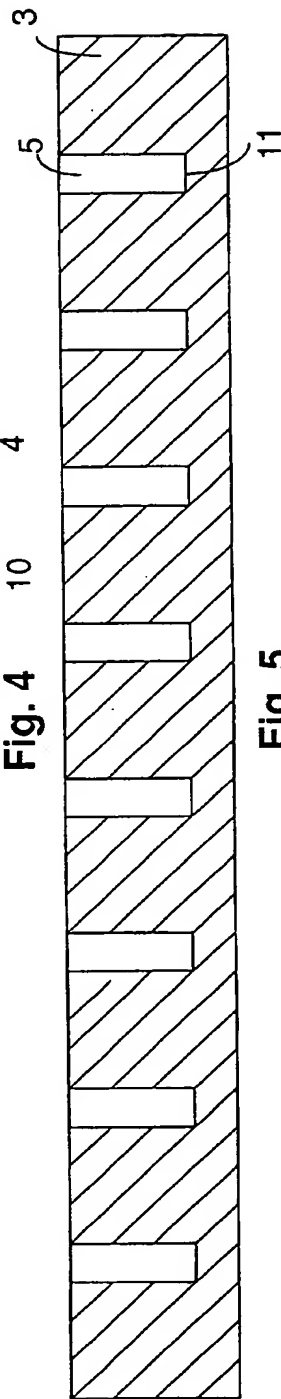


Fig. 5

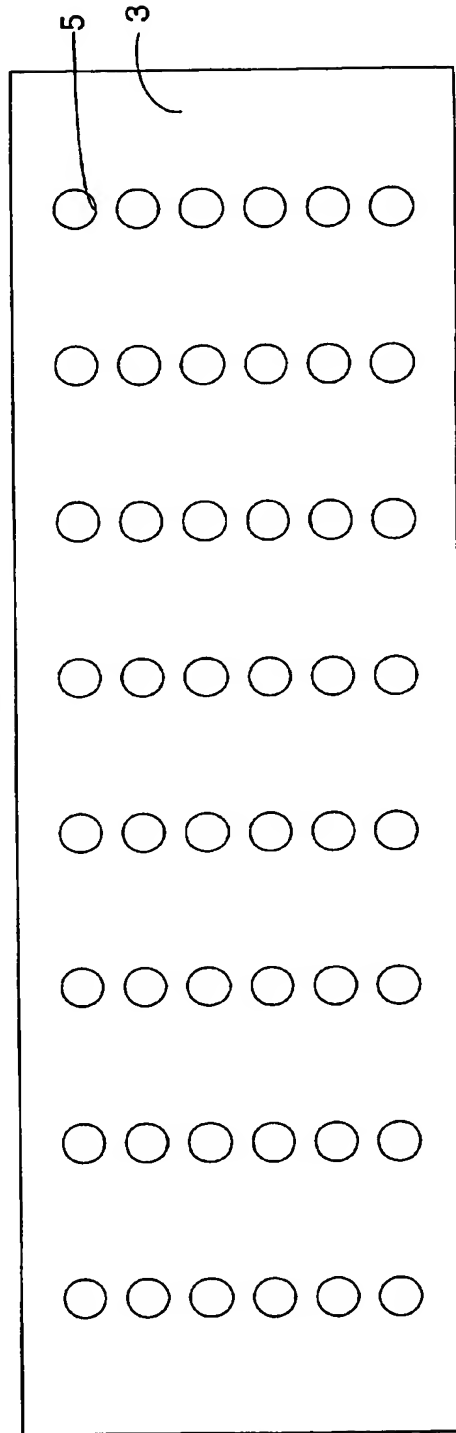


Fig. 6

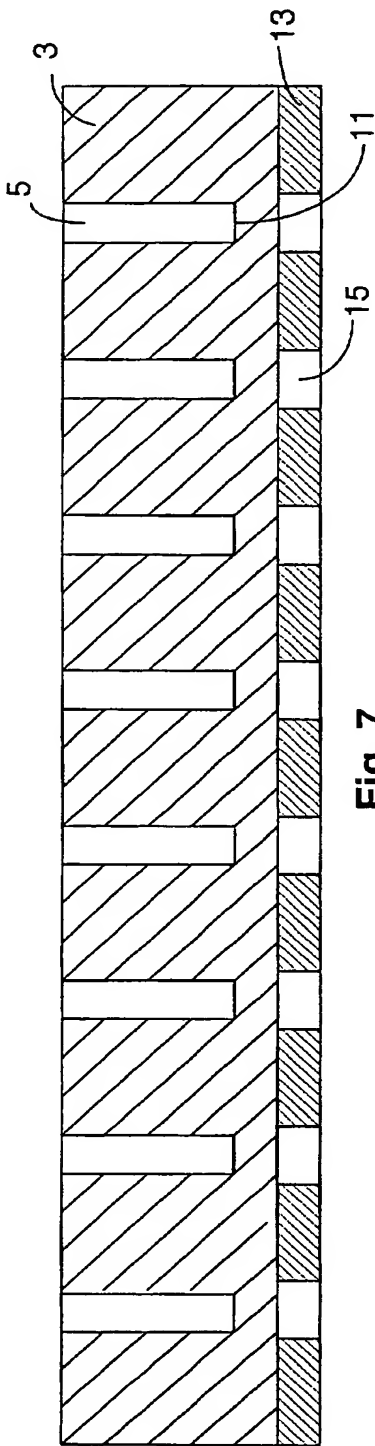


Fig. 7

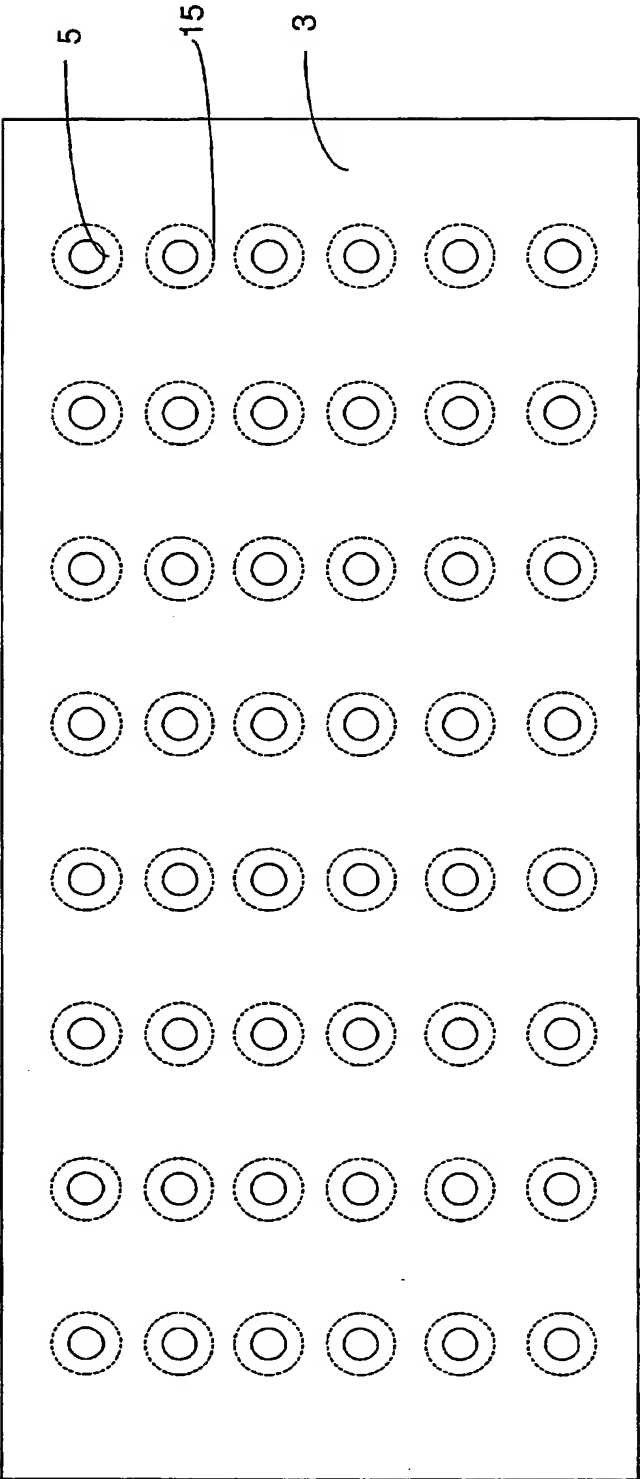


Fig. 8